

1. Rigid-Chain Polymers: Aromatic Polyamides, Heterocyclic Rigid-Rod Polymers and Polyesters

Doetze J. Sikkema, *Adv. Industr. And Engng Polym. Research* (2022) 5, 80-89

A review of preparation, spinning and properties of high performance fibers based on the title polymers. Chapter 4 in a high performance fiber review.

2. Polymers comprising sulfonated 2,6-diphenyl-1,4-phenyleneoxide repeating units

Doetze Jakob Sikkema

From *PCT Int. Appl.* (2019) [WO 2019009721A1 20190110](#), Language: English

The invention relates to polymers comprising sulfonated 2,6-diphenyl-1,4-phenyleneoxide repeating units, to a method for their prepn., and to their use in a membrane electrode assembly, in a proton exchange membrane, in a fuel cell, in an electrolyser, in an electrolytic hydrogen compressor or in a flow battery. The invention further relates to a proton exchange membrane comprising said polymer and to a method for the prepn. Of a proton exchange membrane from said polymer. The invention also relates to the use of the polymers in ion exchange materials.

3. An all-aromatic polypyridine: Monomer and polymer synthesis; Film formation and crosslinking; A candidate fuel cell membrane

Doetze J. Sikkema, Ron M. Versteegen, Maarten J. Pouderoijen, Henk M. Janssen, Ben Boere, Ferry Brands, GerJan Kemperman, Jos B.M. Rewinkel, Menno Koeman

Journal of Power Sources (2018) 379, 234-239

Monomer and polymer synthesis up to kg scale are described; membrane casting and evaluation in high temperature PEMFC are discussed..

4. Process for crosslinking a polymer comprising pyridyl groups

By Sikkema, Doetze Jakob

From *U.S. Pat. Appl. Publ.* (2014), [US 20140243487 A1 20140828](#), Language: English, Database: CAPLUS

The present invention relates to process for crosslinking an arylene oxide polymer comprising a 3-pyridyl and/or a 4-pyridyl group, wherein the polymer is reacted with a compd. according to Formula (1) or a compd. according to Formula (2), wherein R₁, R₂, R₃ and R₄ are independently selected from the group consisting of hydrogen, halogen, hydroxy and cyano. The crosslinked arylene oxide polymer can be used as a membrane, in particular a proton conducting membrane.

5. Phenol compounds and (co)polymers thereof

By Sikkema, Doetze Jakob; Versteegen, Ronny Mathieu; Pouderoijen, Maarten Jozef From *U.S.* (2012), [US 8299202 B2 20121030](#), Language: English, Database: CAPLUS

The present invention relates to A phenol compd. according to Formula (I): wherein: R₁ is selected from the group consisting of, optionally substituted, 2-pyridyl, 3-pyridyl and 4-pyridyl groups, wherein R₁ is at position 2 or 3 of the phenol ring; R₂ is selected from the group consisting of, optionally substituted, 2-pyridyl, 3-pyridyl, 4-pyridyl and Ph groups, wherein R₂ is at position 5 or 6 of the phenol ring; and the phenol ring is optionally substituted at one or two positions, independently selected from positions 2, 3, 5 and 6, with a halogen atom or a with an optionally substituted C₆- C₁₂ aryl group or an optionally substituted C₁-C₁₀ alkyl group. The present invention relates also to (co)polymers comprising the phenol compd. according to Formula (I) and membranes and ionic resins comprising said (co)polymers.

6. Use of a (1>3)-β-D-glucan as an emulsion stabiliser

By Lommerts, Bert Jan; Nederpel, Quirinus Adrianus; Sikkema, Doetze Jakob; Peeters, Joris Wilhelmus From *U.S.* (2012), [US 8197589 B2 20120612](#), Language: English, Database: CAPLUS

The present invention relates to the use of a (1[®]3)-b-D-glucan as an emulsion stabilizer. The present invention further relates to emulsions comprising a (1[®]3)-b-D-glucan in an amt. of 0.01 to 10 wt. %, based on the total wt. of the emulsion. The present invention also relates to bitumen binder compns. comprising a (1[®]3)-b-D-glucan in an amt. of 0.005 to less than 0.1 wt. %, based on the total wt. of the bitumen binder compn. The present invention further relates to emulsions comprising a novel biodegradable emulsifying agent, in particular in combination with a (1[®]3)-b-D-glucan.

7. Process for removing cations from polyareneazole fiber

By Sikkema, Doetze Jakob

From [U.S. \(2011\), US 7906613 B2 20110315](#), Language: English, Database: CAPLUS

The present invention relates to processes for hydrolyzing polyphosphoric acid in a fiber and the removal of hydrolyzed polyphosphoric acid from the fiber.

8. Process for hydrolyzing polyphosphoric acid in a spun yarn

By Allen, Steven R.; Moore, Steven D.; Newton, Christopher W.; Rodini, David J.; Sikkema, Doetze Jakob

From [U.S. \(2011\), US 7906615 B2 20110315](#), Language: English, Database: CAPLUS

The present invention relates to processes for hydrolyzing polyphosphoric acid in a fiber and the removal of hydrolyzed polyphosphoric acid from the fiber.

9. Liquid crystal main-chain polymers for high-performance fiber applications

By Picken, S. J.; Sikkema, D. J.; Boerstoel, H.; Dingemans, T. J.; van der Zwaag, S.

From [Liquid Crystals \(2011\), 38\(11-12\), 1591-1605](#). Language: English, Database: CAPLUS, DOI:10.1080/02678292.2011.624367

A review. The chem. and physics of high-performance fiber spinning based on main-chain liq. crystal polymer (MCLCP) solns. and melts is discussed, which is the largest industrial application of liq. crystal technol. The high modulus and strength of liq. crystal polymer-based high-performance fibers is due to the exceptionally high orientational order that can be achieved, reaching values of 0.95 and higher. Together with the chem. that ensures strong intermol. interactions, often based on H bonding, it is possible to make fibers with unusual mech. and thermal properties. The modulus and strength of such fibers can reach values at ~50-75% of the theor. limit. Within materials science high-performance fibers are esp. interesting, as they are one of the few systems where the material properties can be successfully predicted based on mol. models for the orientational order together with rather simple assumptions on the effect of flow on the director alignment. The most studied MCLCP systems for high-performance fiber spinning PPTA/H₂SO₄, cellulose/H₃PO₄, PIPD(M5)/PPA and melt-spun Vectran™ are discussed.

10. Use of a (1[®]3)-|3-d-glucan as an emulsion stabilizer for bitumen binder emulsions

By Lommerts, Bert Jan; Nederpel, Quirinus Adrianus; Sikkema, Doetze Jakob; Peeters, Joris

Wilhelmus From [PCT Int. Appl. \(2009\), WO 2009113854 A1 20090917](#), Language: English, Database: CAPLUS

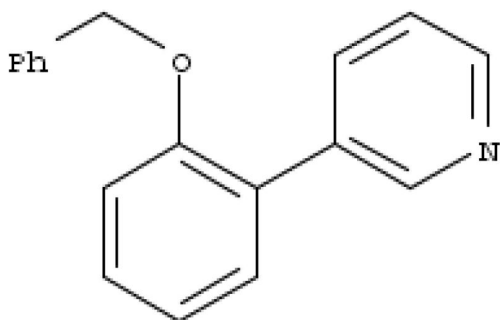
Bitumen emulsions comprises 0.005-0.10 wt.% of a (1[®]3)-b-D-glucan as emulsion stabilizer and a biodegradable emulsifying agent (such as a betaine ester and/or a choline ester) in combination with the (1[®]3)-b-D-glucan (such as scleroglucan).

11. Novel phenol compounds, (co)polymers and membranes

By Sikkema, Doetze Jakob; Versteegen, Ronny Mathieu; Pouderoijen, Maarten Jozef

From [PCT Int. Appl. \(2009\), WO 2009096786 A1 20090806](#), Language: English, Database: CAPLUS

The phenol compd. has formula R₁R₂C₆H₃OH, where R₁ = optionally substituted, 2-pyridyl, 3-pyridyl and 4-pyridyl groups, where R₁ is at position 2 or 3 of the phenol ring; R₂ = optionally substituted, 2-pyridyl, 3-pyridyl, 4-pyridyl and Ph groups, where R₂ is at position 5 or 6 of the phenol ring; and the phenol ring is optionally substituted at 1 or 2 positions, independently selected from positions 2, 3, 5 and 6, with a halogen atom or with an optionally substituted C₆-C₁₂ aryl group or an optionally substituted C₁-10-alkyl group. The (co)polymers comprising the phenol compd. are used to fabricate membranes and ionic resins comprising the (co)polymers. Polymer was prepd. of 2,6-bis(3-pyridyl)phenol (prepn. given), h 0.35 g/dL (0.1% in 95% H₂SO₄).



12. High performance fibers based on rigid and flexible polymers

By Afshari, Mehdi; Sikkema, Doetze J.; Lee, Katelyn; Bogle, Mary
 From [Polymer Reviews \(Philadelphia, PA, United States\) \(2008\), 48\(2\), 230-274](#). Language: English, Database: CAPLUS, DOI:10.1080/15583720802020129

A review. This chapter covers recent developments in the prodn. of well established high performance fibers such as Kevlar, PBO, Spectra and Dyneema fibers and depicts a new super strong M5. The latter fibers have the modulus of 330GPa and tenacity of 5GPa. DuPont de Nemours is currently developing com. M5 fibers and yarns. A very interesting monomer namely, 2,5-dihydroxyterephthalic is used for making poly{2,6-diimidazo[4,5-b:4',5'-e]pyridinylene-1,4-(2,5- dihydroxy)phenylene} (PIPD). The unique feature of the polymer is that the two hydroxyl groups (on terephthalic acid) can form intermol. hydrogen bonds and therefore fibrillation, that is often a problem for aramid fibers, is practically eliminated. As a result, M5 fibers have the highest compressive strength among synthetic fibers. Exploratory evaluation of the UV stability of M5 indicated excellent performance in that field. The mech. properties of the new fiber make it competitive with carbon fiber in most applications - in light, slender, load bearing stiff advanced composite components and structures.

13. A rigid rod polypyridobisimidazole: "M5", towards a new generation of structural composites

By Sikkema, Doetze J.
 From [Proceedings of the Aachen-Dresden International Textile Conference \(2007\), 1st, 36-56](#). Language: English, Database: CAPLUS

A review. In a quest to create polymers as rigid-rod like as PBO, with strong intermol. hydrogen bonds, a novel polymer, formed from 2,3,5,6-tetraaminopyridine and 2,6-dihydroxyterephthalic acid, routinely called 'M5' appears to meet the bill. In fact, great improvement on the synthesis routes has been achieved. The mech. properties of the new fiber make it competitive with carbon fiber in most applications. It is also easy to fabricate into composite form and possess high damage tolerance.

14. Process for the production of polyareneazole yarn

By Allen, Steven R.; Moore, Stephen D.; Newton, Christopher William; Rodini, David J.; Sikkema, Doetze Jakob
 From [PCT Int. Appl. \(2006\), WO 2006135470 A2 20061221](#), Language: English, Database: CAPLUS

The present invention concerns a process for making a polyareneazole multifilament yarn comprising: (a) extruding a soln. comprising polyareneazole polymer and polyphosphoric acid through a plurality of orifices to produce filaments; (b) forming a multifilament yarn from the filaments; (c) hydrolyzing at least some of the polyphosphoric acid in the yarn by heating the yarn to a temp. above about 120 OC for up to about two minutes; (d) washing at least some of the hydrolyzed polyphosphoric acid from the yarn; (e) drying the washed yarn; (f) optionally, heating the yarn above about 300 OC, and (g) collecting the yarn at a speed of at least about 50 m per min.

15. Polyareneazole polymer fibers having pendant hydroxy groups and cations

By Sikkema, Doetze, Jakob
 From [PCT Int. Appl. \(2006\), WO 2006105232 A1 20061005](#), Language: English, Database: CAPLUS

The fibers comprise a polyareneazole polymer having pendant hydroxy groups and >2% based on fiber wt. of cations including Na, K, Ca or any combination thereof. The polyareneazole is a polypyridazole, or more particularly, a polypyridobisimidazole.

16. Processes for preparing monomer complexes and rigid-rod polyareneazole polymers useful for filaments and yarns

By Sikkema, Doetze Jakob; Rodini, David J.; Dindi, Hasan; Schultz, James Arnold; Martin, Georg Valentin; Demuth, Ralf; Schelhaas, Michael
 From [PCT Int. Appl. \(2006\), WO 2006105076 A2 20061005](#), Language: English, Database: CAPLUS

Disclosed are processes for prepreg. monomer complexes that include contacting 2,3,5,6-tetraaminopyridine free base

in water with 2,5-dihydroxyterephthalic acid dipotassium salt to form an aq. mixt., and adjusting the pH of the aq. mixt. to within the range of from about (3) to about (5) to ppt. the monomer complex. Processes of polymg. the monomer complexes, polyareneazoles, filaments and yarns are also disclosed.

17. Process for hydrolyzing polyphosphoric acid in spun yarns

By Allen, Steven R.; Moore, Stephen D.; Newton, Christopher W.; Rodini, David J.; Sikkema, Doetze Jakob From [PCT Int. Appl. \(2006\), WO 2006105226 A1 20061005](#), Language: English, Database: CAPLUS

A process for removing polyphosphoric acid (PPA) from fibers comprises the steps of (a) heating a fiber comprising a polymer and PPA to $>120^{\circ}$ for a time effective to hydrolyze PPA; and (b) in a sep. step, removing hydrolyzed PPA from the fiber with a fluid having a temp. of $<100^{\circ}$. The polymer comprises a polyareneazole such as poly(2,6-diimidazo[4,5-b:4',5'-e]pyridinylene-1,4-(2,5-dihydroxy)phenylene). The heating includes convective heating, radiant heating, radiation heating, RF heating, conductive heating, steam heating, or any combination thereof. Removing hydrolyzed PPA includes washing the fiber with a base.

18. Increasing polymer inherent viscosity, high viscosity rigid rod polymer compositions suitable for spinning into filaments

By Sikkema, Doetze, Jakob; Rodini, David, J.; Adkins, Qinghong, Fu; Allen, Steven, R.; Martin, Georg, Valentin; Demuth, Ralf; Schelhaas, Michael

From [PCT Int. Appl. \(2006\), WO 2006105080 A1 20061005](#), Language: English, Database: CAPLUS

Processes for prepg. polyareneazole polymers include contacting a molar excess of a free base in H₂O with a terephthalic acid salt to form an aq. mixt., adjusting the pH to ppt. a monomer complex, contacting the monomer complex with metal powder, and polymg. the monomer complex. Polyareneazoles, filaments and yarns are also disclosed.

19. Processes for preparing high inherent viscosity polyareneazoles using metal powders

By Sikkema, Doetze Jakob; Rodini, David J.; Adkins, Qinghong Fu; Allen, Steven R.

From [PCT Int. Appl. \(2006\), WO 2006105229 A1 20061005](#), Language: English, Database: CAPLUS

Disclosed are processes for prepg. polyareneazole polymers that include contacting, in polyphosphoric acid, azole-forming monomers and metal powder, the metal powder added in an amt. of from about 0.05 to about 0.9 %, based on the total wt. of the azole-forming monomers, and reacting the azole-forming monomers to form the polyareneazole polymers. Polyareneazoles, filaments and yarns are also disclosed.

20. High inherent viscosity polyareneazoles and fibers therefrom

By Sikkema, Doetze Jakob; Rodini, David J.; Adkins, Qinghong Fu; Allen, Steven R.; Newton, Christopher William From [PCT Int. Appl. \(2006\)](#), [WO 2006105228 A1 20061005](#), Language: English, Database: CAPLUS

Disclosed are processes for prepg. polyareneazole polymers characterized as providing polymer solns. having an inherent viscosity of at least about 22 dL/g at 30° at a polymer concn. of 0.05 g/dL in methane sulfonic acid. Polyareneazoles, filaments and yarns are also disclosed.

21. Process for removing cations and polyphosphoric acid from polyareneazole fiber

By Sikkema, Doetze Jakob

From [PCT Int. Appl. \(2006\)](#), [WO 2006105078 A1 20061005](#), Language: English, Database: CAPLUS

A process for removing cations (and P) from a polyareneazole fiber comprises the steps of (a) providing a fiber comprising a polyareneazole polymer having pendant hydroxy groups and >2% by wt. cations, (b) contacting the fiber with an aq. soln. contg. acid to release at least a portion of the cations, and (c) optionally, washing the fiber with water.

22. Preparation of polyareneazoles and their use in fibers

By Adkins, Qinghong Fu; Sikkema, Doetze Jakob; Rodini, David J.

From [PCT Int. Appl. \(2006\)](#), [WO 2006105227 A1 20061005](#), Language: English, Database: CAPLUS

A process for making polyareneazole comprises: contacting a complex comprising arom. or heteroarom. tetraamine and arom. or heteroarom. diacid in a dehydrating solvent; wherein each amine group of the tetraamine is positioned on the arom. or heteroarom. ring moiety adjacent to at least one of the remaining amine groups of the tetraamine, and wherein the acid groups of the diacid are bonded to non-adjacent arom. or heteroarom. ring carbon atoms of the diacid; and (a) heating with agitation to a temp. of about 90-110° for 30 to 100 min; (b) heating the product of step (a) with agitation to 130-145° for 75 to 300 min; (c) heating the product of step (b) with agitation to 175-185° for 40 to 90 min; and (d) heating the product of step (c) with agitation to 186-220° for 15 to 90 min to provide the polyareneazole.

23. Process for production of polyareneazole polymers

By Allen, Steven R.; Moore, Stephen D.; Newton, Christopher William; Rodini, David J.; Adkins, Qinghong Fu; Sikkema, Doetze Jakob

From [PCT Int. Appl. \(2006\)](#), [WO 2006104974 A1 20061005](#), Language: English, Database: CAPLUS

A process for making a rigid-rod polyareneazole polymer comprises the steps of (a) contacting azole-forming monomers (preferably 2,3,5,6-tetraaminopyridine and 2,5-dihydroxyterephthalic acid), metal powder, and optionally P₂O₅, in polyphosphoric acid to form a mixt.; (b) blending the mixt. at 50-110°; (c) further blending the mixt. at a temp. of up to 145° to form a soln. comprising an oligomer; (d) optionally, degassing the soln.; and (e) reacting the oligomer soln. at 160-250° for a time sufficient to form a polymer. The polymer is used for manuf. of filaments and yarns.

24. Process for removing phosphorous from fibers or yarns

By Allen, Steven, R.; Sikkema, Doetze Jakob

From *PCT Int. Appl.* (2006), [WO 2006105225 A1 20061005](#), Language: English, Database: CAPLUS

A process for removing phosphorus from a yarn (contg. 1.5% P) spun from a polymer soln. contg. polyphosphoric acid comprises contacting the yarn with a base and washing the yarn with an aq. fluid contg. an acid. The polymer comprises a polyareneazole, or more particularly, a polypyridobisimidazole.

25. Production of aromatic heterocyclic rod fibers or films with high tensile strength and modulus

By Sikkema, Doetze Jakob; Northolt, Maurits Gerhard; Zegers, Hendrikus Cornelis

From *Eur. Pat. Appl.* (2006), [EP 1614778 A1 20060111](#), Language: English, Database: CAPLUS

A process for obtaining an arom. heterocyclic rod fiber or film having high tensile strength and/or modulus comprises spinning a synthetic org. polymer to an arom. heterocyclic rod fiber or obtaining the synthetic org. polymer as an arom. heterocyclic rod film, followed by loading the fiber or film in the presence of a processing aid at a temp. below the b.p. of the processing aid and above -50° and at a tension of 10-95% of the fiber or film breaking strength, followed by removing the processing aid and/or performing a heating step at a tension of 10-95% of the fiber or film breaking strength. The method provides a substantial increase of tensile strength (by a factor of > 2) and modulus of arom. heterocyclic rod fibers or films, such as polybenzoxazole (Zylon), and polybenzimidazole (M 5).

26. Assessment of new high-performance fibers for advanced applications

By Sikkema, Doetze J.; Northolt, Maurits G.; Pourdeyhimi, Behnam

From *MRS Bulletin* (2003), [28\(8\), 579-584](#). Language: English, Database: CAPLUS, DOI:10.1557/mrs2003.169

A review. High-performance fibers, used in fabric applications ranging from bulletproof vests to trampolines, must have a sufficient no. of chem. and phys. bonds for transferring the stress along the fiber. To limit their deformation, the fibers should possess high stiffness and strength. Stiffness is brought about by the degree to which the chem. bonds are aligned along the fiber axis. In fiber-reinforced composites, the fibers are the load-bearing element in the structure, and they must adhere well to the matrix material. An ideal reinforcing fiber must have high tensile and compressive moduli, high tensile and compressive strength, high damage tolerance, low sp. wt., good adhesion to the matrix materials, and good temp. resistance. This article reviews and compares the properties and behavior of novel high-performance fiber materials including polyethylene, aramid polybenzobisoxazole, M5, and carbon fibers.

27. NMR based determination of minute acid functionality: end-groups in PET

By Ma, Y.; Agarwal, U. S.; Vekemans, J. A. J. M.; Sikkema, D. J.

From *Polymer* (2003), [44\(16\), 4429-4434](#). Language: English, Database: CAPLUS, DOI:10.1016/S0032-3861(03)00402-6

Conditions are detd. for the carbodiimide-mediated room temp. esterification of the carboxylic acid end-groups of poly(ethylene terephthalate) with hexafluoroisopropanol. The hexafluoroisopropyl ester is quantified with ¹⁹F NMR, using a,a,a-trifluorotoluene as a secondary std. This provides a technique for accurate detn. of minute amts. of carboxylic acid functionality in small samples of polymers, and potentially in animal- and plant-based foods.

28. Solid-state polymerization of PET: influence of nitrogen sweep and high vacuum

By Ma, Y.; Agarwal, U. S.; Sikkema, D. J.; Lemstra, P. J.

From *Polymer* (2003), [44\(15\), 4085-4096](#). Language: English, Database: CAPLUS, DOI:10.1016/S0032-3861(03)00408-7

The authors have examd. the influence of reaction environment on the solid-state polymn. (SSP) of thin (180 pm) poly(ethylene terephthalate) (PET) chips at 250° by following the intrinsic viscosity (IV) increase and the end-group depletion. When the SSP reaction is carried out in vacuum, IV increases from 0.58 to 2.4 dL/g in 2.5 h of reaction. The initially rapid reaction slows considerably with time, and IV rise nearly stops at 2.75 dL/g at 6 h, though the authors still detect the acid and the hydroxyl end-groups at concns. of 3 and 5 mequiv./kg, resp. This suggests a role of crystn. in limiting the approachability of the end-groups to each other, thereby temporarily rendering them inactive. At this stage, raising the temp. to 270° to melt the PET in vacuum again increases the IV to 2.97 dL/g in 1.5 h, perhaps due to the release of cryst. restraints in the melt allowing some these inactive end-groups to approach each other. They find that accounting for these temporarily inactive end-groups is a must for a good kinetic description of SSP to IV > 1.3 dL/g. When nitrogen is used as a carrier gas, the reaction rate and the extent of mol. wt. build-up are somewhat lower compared to SSP under vacuum. A sublimate is collected during SSP under vacuum, and the authors find it to be made up of terephthalic acid, monohydroxyethyl terephthalate, bishydroxyethyl terephthalate, and cyclic oligomers. This indicates the presence of a new condensation mechanism during SSP under vacuum.

29. PIPD, a new high-modulus and high-strength polymer fibre with exceptional fire protection properties

By Northolt, M. G.; Sikkema, D. J.; Zegers, H. C.; Klop, E. A.

From [Fire and Materials \(2002\), 26\(4-5\), 169-172](#). Language: English, Database: CAPLUS, DOI:10.1002/fam.793

The development of the new high-modulus and high-strength fiber M5, made of poly{2,6-diimidazo[4,5-b:4',5'-e]pyridinylene-1,4(2,5-dihydroxy)phenylene} or PIPD, has resulted in an org. polymer fiber with exceptional fire protection properties when compared with PBO, Twaron, Kevlar and Nomex fibers. The PIPD as-spun fiber with a modulus of 150 GPa and a tensile strength of 2.5 GPa is a crystal hydrate contg. 21 wt% water. Cone calorimeter measurements yielded a fire performance index (FPI) 20 times higher than Nomex, with extremely low value for the specific extinction area (SEA) characterizing the smoke formation. The PIPD-HT fiber (recrystd. in an after treatment into the anhyd. cryst. structure) with a modulus > 300 GPa and a strength > 5 GPa has a FPI value similar to PBO but shows much less smoke formation than this fiber.

30. Other high modulus-high tenacity (HM-HT) fibres from linear polymers

By Beers, David; Young, Robert J.; So, C. L.; Sikkema, Doetze J.; Perepelkin, Kirill E.; Weedon, Gene Edited By:Hearle, J. W. S

From [High-Performance Fibres \(2000\), 93-155](#). Language: English, Database: CAPLUS, DOI:10.1533/9781855737549.93

A review on the prodn., properties, and applications of high modulus-high tenacity fibers from linear polymers.

31. Manmade fibers one hundred years: polymers and polymer design

By Sikkema, Doetze J.

From [Journal of Applied Polymer Science \(2002\), 83\(3\), 484-488](#). Language: English, Database: CAPLUS, DOI:10.1002/app.2254

A review. A brief overview of a century of manmade fiber development (particularly tech., i.e., strong and high E-modulus, fibers), emphasizing the last quarter century where the work increasingly grew in the direction of rational design of new structures, as the understanding of what makes a fiber do its job well grew.

32. Synthesis and structure of a new polyalcohol

By Lommerts, Bert Jan; Sikkema, Doetze J.

From [Macromolecules \(2000\), 33\(21\), 7950-7954](#). Language: English, Database: CAPLUS, DOI:10.1021/ma991262s
A new polyalc. is prepd. by borohydride redn. of the perfectly alternating ethylene-carbon monoxide copolymer. The low melting temp. of this atactic material (137 ° C) is attributed to the 1,4-arrangement of the hydroxyl side groups. The different orientations of these slightly bulky groups with respect to the mol. plane give rise to less efficient packing of the chains in the crystal lattice. The reflections in the X-ray diffraction pattern are indexed on the basis of an orthorhombic lattice. The derived unit cell dimension are a = 8.78 Å, b = 5.47 Å, and c = 7.47 Å, and the cryst. d. (1.08 g/cm³) is significantly less than the cryst. d. of atactic poly(vinyl alc.) (1.35 g/cm³), which has a 1,3-arrangement of the hydroxyl substituents. Soln. cast polyalc. films could be drawn at 110 ° C to a ratio of about 10; a tensile strength of 0.5-0.6 GPa and a (max.) tensile modulus of 11 GPa are attained.

33. Flame retardant materials

By Northolt, Maurits Gerhard; Sikkema, Doetze Jakob

From [PCT Int. Appl. \(1999\), WO 9927169 A1 19990603](#), Language: English, Database: CAPLUS

Flame retardant materials prep'd. from hydrates of polymers can be used for manuf. of flame retardant materials, such as composites, fabrics, nonwovens, films, foams, and paper, with a significant improvement with respect to the non-hydrated materials. Preferred are hydrates of hydroxy-contg. polymers, and more preferably fully hydrated rigid rod polymers, in particular fully hydrated PIPD (polypyridobisimidazole) fibers.

34. Mechanical properties and structural transitions in the new rigid-rod polymer fiber PIPD ('M5') during the manufacturing process

By Lammers, M.; Klop, E. A.; Northolt, M. G.; Sikkema, D. J.

From [Polymer \(1998\), 39\(24\), 5999-6005](#). Language: English, Database: CAPLUS, DOI:10.1016/S0032-3861(98)00021-4

The polymer poly[2,6-diimidazo[4,5-b:4',5'-e]pyridinylene-1,4-(2,5-dihydroxy)phenylene] (PIPID) is spun from a liq. cryst. soln. in polyphosphoric acid. The as-spun fiber is a crystal hydrate, which transforms into a bidirectional hydrogen-bonded structure during heat treatment. This transition results in an increase in cryst. modulus along the chain direction due to a decrease of the cross-sectional area per chain, and in an increase in shear modulus due to stronger interaction between the chains caused by the hydrogen bonds. Heat-treated PIPID has a tensile modulus of up to 360 GPa, a tensile strength well above 4 GPa, an internal shear modulus of 7 GPa, and a compressive strength of 1.7 GPa.

35. Design, synthesis and properties of a novel rigid rod polymer, PIPD or "M5": high modulus and tenacity fibers with substantial compressive strength

By Sikkema, Doetze J.

From [Polymer \(1998\), 39\(24\), 5981-5986](#). Language: English, Database: CAPLUS, DOI:10.1016/S0032-3861(97)10289-0

A rigid rod polymer with strong hydrogen bonds between the polymer chains was found in a polypyridobisimidazole based on 2,3,5,6-tetraaminopyridine and 2,5-dihydroxyterephthalic acid. The strong interchain forces translate into the highest compressive strength found in polymer fibers by far, and the polarity of the polymer seems to contribute to easy adhesion in advanced composite manuf. The modulus and tenacity are as expected for a rigid rod polymer in the general class of the earlier PBZT and PBO fibers.

36. Process for dicarboxylating dihydric phenols

By Sikkema, Doetze Jakob; Reichwein, Adrianus Maria

From [PCT Int. Appl. \(1997\), WO 9717315 A1 19970515](#), Language: English, Database: CAPLUS

A process for dicarboxylating dihydric phenols, particularly for prepg. 2,5-dihydroxyterephthalic acid, is described where the dihydric phenol (e.g., hydroquinone) is reacted with CO₂ in the presence of an alkali metal carbonate (e.g., K₂CO₃), the reaction being carried out in the presence of alkali metal formate (e.g., HCO₂K) at a temp. above the formate's m.p. This process has advantages over the Kolbe-Schmitt reaction and other known carboxylation reactions such using a lower operating pressure, giving higher yields, and requiring shorter reaction times.

37. Nitration of pyridine-2,6-diamines

By Sikkema, Doetze Jacob

From [PCT Int. Appl. \(1997\), WO 9711058 A1 19970327](#), Language: English, Database: CAPLUS

It has been found that the nitration of pyridine-2,6-diamines, which is commonly performed in a mixt. of nitric acid and conc'd. sulfuric acid, will give a significantly higher yield (from about 50% up to more than 90%) if the reaction is carried out in a mixt. of nitric acid and oleum, or by otherwise providing an inherently anhyd. medium.

38. Preparation of pyridine-2,6-diamines from 3-hydroxyglutaronitrile and amines.

By Reichwein, Adrianus Maria; Sikkema, Doetze Jakob

From [PCT Int. Appl. \(1996\), WO 9636607 A1 19961121](#), Language: English, Database: CAPLUS

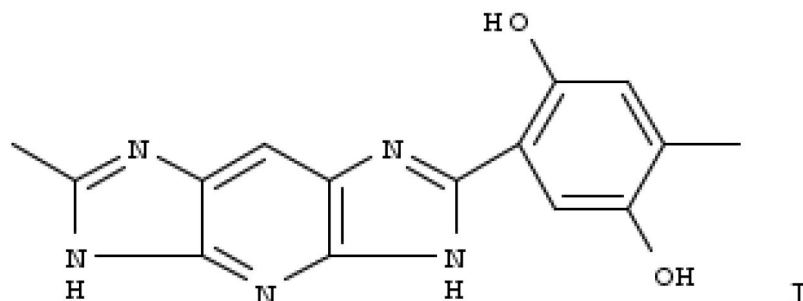
Pyridine-2,6-diamines having >1 primary amino group were prepd. by reaction of 3-hydroxyglutaronitrile (I) with NH₃ (donors), or primary or secondary amines. In the case of a primary or secondary amine, no hydrogen halide catalyst is used. Thus, I, NH₃, and CuCl in MeOH were heated at 150° in an autoclave for 3 h to give >95% 2,6-diaminopyridine.

39. Rigid-rod polymer based on pyridobisimidazole, its preparation, its spinnable solutions, and fibers therefrom

By Sikkema, Doetze Jakob; Lishinsky, Vadim Leonidovich

From [PCT Int. Appl. \(1994\), WO 9425506 A1 19941110](#), Language: English, Database: CAPLUS

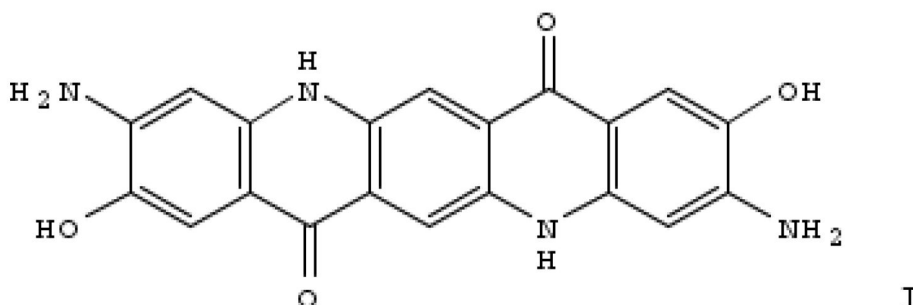
A rigid rod polymer has >50% of the recurring groups satisfying the formula I; in the remaining groups the 2,5-dihydroxy- p-phenylene may be replaced by an arylene, which may be substituted or not, and the pyridobisimidazole may be replaced by benzobisimidazole, benzobisthiazole, benzobisoxazole, pyridobisthiazole, and/or pyridobisoxazole. A 10-21 wt.% soln. of the polymer in polyphosphoric acid can be spun into fibers having exceptional properties.



40. One-pot synthesis of a pentacyclic bis(aminophenolic)bipyridone from monocyclic precursors

By Sikkema, Doetze J.

From [Synthesis \(1993\), \(12\), 1211-12](#). Language: English, Database: CAPLUS, DOI:10.1055/s-1993-26025
1,4-Cyclohexanedione-2,5-dicarboxylic Me ester can be condensed with 2,4-diaminophenol, aromatized and cyclized to 3,10-diamino-2,9-dihydroxyquino[2,3-b]acridine-7(5H),14(12H)-dione I in a one-pot procedure. The product must be handled with care to avoid oxidn.; isolation as the hydrochloride salt allows convenient manipulation and purifn. to polymn. quality material.



41. High-temperature, solvent-resistant amorphous matrix materials and their thermoplastic composites

By Sikkema, Doetze J.; Middelman, Erik

From [Polymer Preprints \(American Chemical Society, Division of Polymer Chemistry\) \(1992\), 33\(1\), 406](#). Language: English, Database: CAPLUS

Bis(4-aminophenyl)methane, isophthaloyl chloride, p-phenylenediamine, and terephthaloyl chloride are copolymd. to form a polyamide. The dynamic mech. properties are discussed.

42. p-Phenylene-2,2'-bis(3-aminopropenenitrile) and its polymerization with terephthaloyl dichloride

By Sikkema, Doetze J.; Reichwein, John

From [Macromolecular Chemistry and Physics \(1994\)](#), 195(1), 273-8. Language: English, Database: CAPLUS, DOI:10.1002/macp.1994.021950125

With the aim of prep. a new rigid rod type polymer, the authors worked out a synthesis of the title compd. (I): 1,4-bis(chloromethyl)benzene was reacted with KCN, and the product was formylated with Et formate; exchange of enol by enamine function by treatment with ammonium acetate completed the synthesis. Polycondensation of the only mildly nucleophilic monomer I with terephthaloyl chloride proceeded rather slowly and only moderate chain length polyamide proved accessible. The cyclization envisioned could be shown to proceed as desired in a model compd. from I with PhCOCl. Upon putative cyclization, the polymer remained sol. in H₂SO₄.

43. One pot synthesis of 2,6-dichloro-3,5-dicyanopyridine from aliphatic precursors

By Duindam, Antoine; Lishinsky, Vadim L.; Sikkema, Doetze J.

From [Synthetic Communications \(1993\)](#), 23(18), 2605-9. Language: English, Database: CAPLUS, DOI:10.1080/00397919308012595

Malononitrile is condensed with tri-Et orthoformate in the presence of pyridine; the mixt. is acidified with HCl gas and, after addn. of further pyridine.HCl, diazotized to form 2,6-dichloro-3,5-dicyanopyridine in a convenient, high yield process.

44. Lyotropic main chain liquid crystal polymers

By Northolt, M. G.; Sikkema, D. J.

Edited By: Collyer, A. A

From [Liq. Cryst. Polym. \(1992\)](#), 273-348. Language: English, Database: CAPLUS

A review with 249 refs. with emphasis on the statistical physics of the title polymers via a modified Maier-Saupe theory developed by Picken.

45. A new amorphous heat-resistant aromatic polyamide fiber prepared by wet-spinning

By Jager, Jan; Krins, Bas; Sikkema, Doetze J.

From [Journal of Applied Polymer Science \(1993\)](#), 48(11), 1945-51. Language: English, Database: CAPLUS, DOI:10.1002/app.1993.070481108

A new amorphous heat-resistant arom. polyamide was prepd. by soln. polymn. in N-methylpyrrolidone (I) from equimolar amts. of 4,4'-methylenedianiline, p-phenylenediamine, isophthaloyl chloride, and terephthaloyl chloride. After neutralization of the byproduct HCl with Ca(OH)₂, the as-polymd. polymer soln. (15.7% polyamide in I) was spun directly on a horizontal wet-spinning device. Yarn drawing of as-spun fibers, near the glass transition temp. of the polymer, was performed on hot-drawing equipment. To det. the optimal drawing conditions, the influence of the draw ratio on the tensile properties was studied in detail. On the basis of these results, a draw ratio of 1.7 was applied to prep. a large quantity of drawn polyamide multifilament yarn (f330 and f500). Tensile properties of both fibers and filaments were detd. by std. methods. The fibers are characterized by an excellent thermal stability as indicated by temp.-dependent tensile measurements, long-term heat resistance, boiling-H₂O shrinkage, and hot-air shrinkage, of the fibers. Despite the amorphous character of the polyamide fiber, it offers an excellent resistance to common org. solvents (except THF and DMF). Moreover, the fiber exhibits a good resistance toward dil. inorg. acids, salts, and bases. Depending on the specific properties of the polyamide fiber, the material may find applications in hot-air filter fabrics, protective clothing, elec. insulation paper, or advanced structural materials.

46. Preparation, morphology, and deformation behavior of poly(oxypropylene-co-nylon 6) thermoplastic elastomers

By Sikkema, D. J.

From [Journal of Applied Polymer Science \(1991\)](#), 43(5), 877-81. Language: English, Database: CAPLUS, DOI:10.1002/app.1991.070430504

Segmented elastomeric copolymers contg. nylon 6 and polyoxypropylene blocks were prepd. and characterized. Pronounced phase sepn. in the solid phase was deduced from DSC, small-angle x-ray scattering (SAXS), and dynamic mech. measurements. The morphol. (from SAXS) and anisotropy in mech. properties strongly suggested phase sepn. in the melt (which was optically clear). Elastomerlike properties were described and discussed.

47. Lyotropic main-chain liquid crystal polymers

By Northolt, M. G.; Sikkema, D. J.

From [Advances in Polymer Science \(1991\), 98\(Sep. Tech. Thermodyn. Liq. Cryst. Polym.\)](#), 115-77.

Language: English, Database: CAPLUS

A review with 230 refs. on the lyotropic liq.-cryst. polymers with emphasis on the synthetic aspects, order in lyotropic solns., morphol. of fibers and films, and mech. and thermal properties.

48. Advances in Polymer Science, 98: Separation Techniques, Thermodynamics, Liquid Crystal Polymers

By Northolt, M. G.; Raetzsch, M. T.; Sikkema, D. J.; Wild, L.; Wohlfahrt, C.

From [No Corporate Source data available \(1991\), 198 pp.](#) Language: English, Database: CAPLUS

49. Matrix materials for rugged composites

By Sikkema, Doetze J.

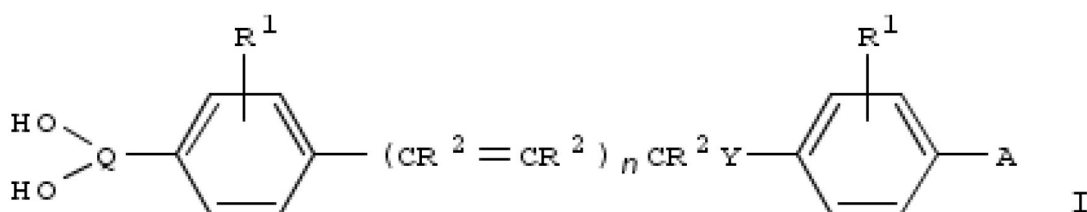
From [CHEMTECH \(1990\), 20\(11\), 688-91](#). Language: English, Database: CAPLUS

A review with 3 refs. on prepn. and properties of high-temp. amorphous arom. polyamides used as matrixes for rugged composites.

50. Polyurethanes suitable for optical waveguides

By Wreesmann, Carel Theodoor Jozef; Erdhuisen, Erwin Wilhelmus Petrus; Sikkema, Doetze Jakob From [Eur. Pat. Appl. \(1990\), EP 350112 A1 19900110](#), Language: English, Database: CAPLUS

The title polyurethanes, with high hyperpolarizability d . (Dh), are prepd. from the diols I [A = CN, NO₂, NCCH:C(CN)₂, C:C(CN)₂, CF₃; Q = aliph. C₂₋₁₀ hydrocarbylidyne contg. alkoxy O or tertiary amine N; R₁ = halogen, R₂, OR₂, COR₂, CO₂R₂, CN, CF₃; R₂ = H, alkyl; Y = CR₂, CCN, N; n = 0-4] and have delocalized p-electron systems to which both electron donor and electron acceptor groups are directly coupled. Thus, 4-(2,3-dihydroxypropoxy)-4'-nitrostilbene was polymd. with isophorone diisocyanate to give a polyurethane with glass temp. 130°, wt.-av. mol. wt. 4000, and Dh 2.15 x 10⁻¹¹ mV.



51. A novel high-temperature thermoplastic

By Sikkema, D. J.

From [Polymeric Materials Science and Engineering \(1989\), 60, 623-4](#). Language: English, Database: CAPLUS

High-temp. injection-moldable thermoplastics were prepd. by melt polycondensation of 3-aminobenzoic acid, 4-aminobenzoic acid, isophthalic acid, and bis(4-aminophenyl)methane. The resulting polyamides had glass temps. >250°, good mech. properties, and excellent solvent resistance.

52. Composites comprising an aramid matrix

By Sikkema, Doetze Jakob

From [Eur. Pat. Appl. \(1989\), EP 307993 A1 19890322](#), Language: English, Database: CAPLUS

Comps. comprising 10-80 vol.% reinforcing fibers and 20-90 vol.% thermoplastic fibers or films consisting of amorphous thermoplastic copolyamides contg. units of >2 dicarboxylic acids mol >2 diamines and having the divalent arom. radicals linked by 1,4-phenylene, 1,5-naphthylene, 2,6-naphthylene, 4,4'-diphenylene, p-C₆H₄XC₆H₄-m (X = CYY₁, O, S, SO₂CO; Y, Y₁ = H, C₁₋₄ alkyl, cy cyclohexyl), 1,3-phenylene, 1,6-naphthylene, 2,7-naphthylene, 3,4,1-diphenylene, m-C₆H₄XC₆H₄-p, and/or m-C₆H₄XC₆H₄-m groups, are useful for forming composites with good processability and good phys. properties. Thus, 25.0 mol p-phenylenediamine was copolymd. with bis(4-aminophenyl)methane 25.0, terephthaloyl dichloride 25.05, and isophthaloyl dichloride 25.05 mol to give a copolyamide (I). Poly(p-phenyleneterephthalamide) (II) fibers were passed through a soln. contg. 12.4% I, treated with an aq. coagulating soln., dried, and blended with I fibers to II fiber-I vol. ratio 1:1, and compression molded at 390° to give a composite with flexural modulus 30,847 MPa, flexural stress at max. load 491.3 MPa, and elongation at rupture 3.7%.

53. A novel high-temperature thermoplastic

By Sikkema, D. J.

From [Materials & Design \(1988\), 9\(6\), 343-4](#). Language: English, Database: CAPLUS, DOI:10.1016/0261-3069(88)90109-4

Amorphous arom. polyamides could be produced by multi-component random copolymn. (e.g., 3-aminobenzoic acid/4-aminobenzoic acid/bis(4-aminophenyl)methane/isophthalic acid system). These materials could be

processed by injection molding and showed attractive properties, such as high strength modulus and solvent resistance in addn. to their high glass temp.

54. Carboxymethyl cellulose with xanthan gum like rheology

By Sikkema, D. J.; Janssen, H.

From [Macromolecules \(1989\), 22\(1\), 364-6](#). Language: English, Database: CAPLUS, DOI:10.1021/ma00191a066

By manipulation of the alky. during CM-cellulose (I) prepn. (aiming at the formation of poorly substituted segments), a product resembling a cellulose-I segmented block copolymer can be made with soln. rheograms closely resembling those of xanthan gum. Such results are obtained by carboxymethylation in aq. iso-ArOH or in a dry phase kneader. Some salt resistance can be achieved, although not on the level of xanthan gum. The new products show a temp. dependence of soln. viscosity comparable with that of xanthan gum but do not show the discontinuity at 60° characteristic of xanthan gum.

55. Thermotropic liquid crystal polymers with flexible chain elements. Synthesis, spinning, and mechanical properties

By Sikkema, Doetze J.

Edited By: Lemstra, P. J.; Kleintjens, L. A

From [Integr. Fundam. Polym. Sci. Technol.--2, \[Proc. Int. Meet. Polym. Sci. Technol., Rolduc Polym. Meet.--2\] \(1988\), 566-70](#). Language: English, Database: CAPLUS

A thermotropic liq.-cryst. polyether prepd. by cationic polymn. of 7-oxabicyclo[2.2.1]heptane-tetrahydrofuran mixts. crystd. immediately when melt spun, preventing any orientation by way of spinning channel shear. Terpolymers incorporating 1-5% ethylene oxide as a crystn. retarder could be spun into yarns showing a high mol. orientation and significant crystallinity, although in all cases drawdown was limited to values <2.4*, independent of extrusion rate, spinning temp., or the use of a hot air zone below the spinneret. The tensile properties of the yarn were disappointing. Similar results were obtained with a 4-hydroxybenzoic acid-hydroquinone-azelaic acid copolymer spun at 300° ; drawdown was limited to 8.8* and the as-spun yarns showed pronounced orientation but no proper crystallinity as an oriented nematic structure seemed to be frozen in. The yarn properties were very poor. All the yarns resisted cold or hot drawing. The disappointing mech. properties were attributed to chain folding, resulting in a pseudo-lamellar structure of the liq.-cryst. melt which cannot form in all-arom. polyamide solns. or all-arom. polyester melts. In the case of the more ordered polyether system, exptl. evidence for lamellar organization was obtained.

56. Amorphous aromatic copolyamide, a process for its preparation and shaped objects

By Sikkema, Doetze Jakob

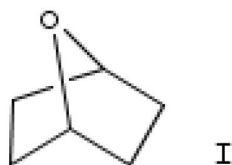
From [Eur. Pat. Appl. \(1987\), EP 239159 A1 19870930](#), Language: English, Database: CAPLUS

Polyamides which can be molded by melt processing without loss of thermal stability comprise repeating units COArCO (A), NHArNH (B), and NHArCO (C) with 17-41.5:17-41.5:17-66 A:B:C, with Ar being arylene radicals. A mixt. contg. bis(4-aminophenyl)methane 1980, 3-aminobenzoic acid 1370, 4-aminobenzoic acid 1370, and isophthalic acid 1660 g was mixed with 4.7 L Ac₂O, heated with stirring to 250° with concurrent removal of AcOH, and heated at 250° in vacuo to give a polyamide (I). I was injection molded (screw temp. 365° , mold temp. 140°) to give a test piece with heat distortion temp. 213° , and glass temp. 235° , vs. 161 and 150, resp., for com. PEEK.

57. An economical and convenient synthesis of 7-oxabicyclo[2.2.1]heptane

By Sikkema, D. J.; Hoogland, P.; Bik, J.; 'T Lam, P.

From [Polymer \(1986\), 27\(9\), 1441-2](#). Language: English, Database: CAPLUS, DOI:10.1016/0032-3861(86)90049-2
A high yield process for converting hydroquinone into 7-oxabicyclo[2.2.1]heptane (I) by hydrogenation over Rh/Al₂O₃, isomerizing the cis-trans mixt. of 1,4-cyclohexanediols into the all trans isomer by treatment with sodium, and ring closure over A4 zeolites was demonstrated on mini-plant scale. Long-term continuous operation of the ring closure reactor gave no problems. The rhodium catalyst is poisoned after short use. Regeneration by mild oxidative treatment was demonstrated for Rh/C catalysts.

**58. Poly(7-oxabicyclo[2.2.1]heptane-co-tetrahydrofuran): polymerization, polymer characterization and spinning**

By Sikkema, D. J.; Hoogland, P.

From [Polymer \(1986\), 27\(9\), 1443-52](#). Language: English, Database: CAPLUS, DOI:10.1016/0032-3861(86)90050-9
A high-yield cationic copolymn. of 7-oxabicyclo[2.2.1]heptane (I) and THF was developed to yield high mol. wts., the mechanism being deduced from product and kinetic studies. The monomer ratios were varied to arrive at a range of products melting at various temps. The crystn. rate was influenced by copolymg. minor amts. of a third monomer-ethylene oxide. Copolymn. proceeded in a truly random fashion, mol. wt. was regulated almost exclusively by pptn. The viscosity of the polymer melt, very high at rest, dropped to a fairly low level upon shearing and the shear stress was essentially independent of the shear rate. The I-THF copolymers [37684-23-4] showed thermotropic liq.-cryst. behavior with as little as 30% of the rigid monomer, I. Spinning expts. were carried out mainly on a miniature spinning app., enabling ram extrusion from an evacuated chamber heated only in the lower part. High mol. orientation was easily achieved in as-spun yarn. Poor drawdown ratios, no higher than 2.4, were obtained. No high-modulus or high-tenacity yarns were achieved. The poor extensibility of the spinning thread seemed to point to extensive chain folding in the (imperfect) lamellae in the liq.-cryst. state.

59. Carboxymethyl cellulose grafted with dextrin: synthesis and solution rheology

By Sikkema, D. J.

From [Journal of Applied Polymer Science \(1985\), 30\(9\), 3523-9](#). Language: English, Database: CAPLUS, DOI:10.1002/app.1985.070300901

Na CM-cellulose (CMC) grafted with dextrin is easily accessible by reacting freshly prepd. CMC with the acid alcoholysis product from starch and glycerol monochlorohydrin [36250-80-3]. The success of the grafting reaction depends on adjusting the alky. of the grafting reaction mass prior to addn. of the dextrin chlorohydrin reagent. In all, less NaOH should be applied than theor. necessary for all Cl substitution. In some cases the products show interesting rheol. at very low shear, but rather CMC-like rheol. or more Newtonian behavior than CMC at shearing rates over a few reciprocal seconds. These results reinforce the credibility of the view that natural polysaccharide gums with unusual rheologies derive their special properties from their well-defined substitution pattern rather than from their overall degree of substitution with (oligo)saccharide units.

60. Flexible layered product

By Vrouwenraets, Cornelius Martinus Franciscus; Sikkema, Doetze Jakob

From [Eur. Pat. Appl. \(1984\), EP 111360 A1 19840620](#), Language: English, Database: CAPLUS

Waterproof garments or tents made by covering a textile material with a waterproof polyester film having water transmission rate >1000 g/m²day which consists of a plurality of recurrent polyester linkages contg. long-chain glycol segments having mol. wt. 800-6000 and carbon-oxygen at. ratio 2.0-4.3:1 and diol residues having mol. wt. <250 . Thus, a mixt. consisting of dimethyl terephthalate 33.3, 1,4-butanediol 21.6, and polyethylene oxide glycol (av. mol. wt. 400) 12.5 g was stirred at 110° , treated with 500 ppm Ti(OBu)₄ (calcd. on dimethyl terephthalate) heated to 160° with distn. of MeOH, reduced in pressure to 100 Pa while heating increased to 245° , continued 3-4 h to give a polyether-polyester [9037-99-4] having relative viscosity 2.53 (1 g in 100 g m-cresol at 25°). The polymer was transformed by film blowing to film having thickness 10-30 μm. The film had water absorption 5% (DIN 53495) and a 12 μm-thick film had a water- vapor permeability 1700 g/m²day. Films laminated with a polyamide 6 fabric were made into garments which had favorable wearability properties.

61. Injection molding objects of high impact strength from a polyamide molding compound

By Cohen, Jacob Louis; Sikkema, Doetze Jakob; Van Berkel, Reinier Willem Marcel

From [Eur. Pat. Appl. \(1982\), EP 60579 A1 19820922](#), Language: English, Database: CAPLUS

The title molding compds. comprise nylon 6 (I) [25038-54-4] or nylon 66 [32131-17-2] contg. a segmented polyamide based on a polypropylene oxide diamine, dicarboxylic acid, and ε-caprolactam(II) and a lubricant. Thus, polypropylene oxide diamine 29,700, adipic acid 2190, II 29,700, heat stabilizer 300, and 85% H₃PO₄ 100 g were heated together 8 h at 225° to give the polyamide [75572-13-3]. Injection molded test bars of I contg. 10% above polyamide and 0.5% Ca stearate [1592-23-0] had Charpy notched impact strength 11.0 kJ/cm² at 23° .

62. Isocyanates from reaction of carbodiimides with carboxylic acids at 280° C

By Van Guldener, Dirk B.; Sikkema, Doetze J.

From [Chemistry & Industry \(London, United Kingdom\) \(1980\), \(15\), 628](#). Language: English, Database: CAPLUS

Reaction of 4-ClC₆H₄CO₂H (I) with (MeC₆H₄N:)₂C gave MeC₆H₄NCOC₆H₄Cl-4 and MeC₆H₄NCO, which was isolated as Bu₂NCONC₆H₄Me by trapping with Bu₂NH. Similar reactions were obsd. for I with [2,6-(Me₂CH)₂C₆H₃N:]₂C.

63. Isotactic poly(allyl vinyl ether). 2. NMR study of stereoregularity and relaxation times

By Angad-Gaur, H.; Sikkema, D. J.

From [Makromolekulare Chemie \(1980\), 181\(11\), 2385-93](#). Language: English, Database: CAPLUS

At a field of 6.34 T, ¹H NMR peaks of poly(allyl vinyl ether) [26747-38-6] overlap severely, hampering a reliable anal. of stereoregularity. In ¹³C NMR, even hexad splittings can be obsd. Relaxation time ratios for backbone C atoms are consistent with dipolar relaxation. The relaxation time for the racemic peak is longer than that for meso peaks, in contrast to data for other polymers. This difference may result from the use of a stronger magnetic field.

64. Isotactic poly(allyl vinyl ether). 1. A study of the polymerization and a proposal of a mechanism of stereocontrol

By Sikkema, D. J.; Angad-Gaur, H.

From [Makromolekulare Chemie \(1980\), 181\(11\), 2259-66](#). Language: English, Database: CAPLUS

Stereospecificity in the polymn. of allyl vinyl ether [3917-15-5] to highly isotactic polymer by SbCl₅ in PhMe is not significantly lower at $\sim -10^\circ$ than at $\sim -75^\circ$, and reproducibility is much better at -10° . NMR anal. (¹H and ¹³C) of dyads, triads, and (partially) hexads gave no evidence for stereocontrol other than the relation between carbocationic chain end and incoming monomer. A mechanism involving only assocn. between monomer and counterion and

involving only steric hindrance is proposed.

65. Improving the adhesion of poly(vinyl chloride) to tissue, texture, fleece, or another poly(methyleneterephthalate) product made of fibers, yarns, or threads

By Sikkema, Doetze Jacob

From *Ger. Offen.* (1980), DE 2932873 A1 19800313, Language: German, Database: CAPLUS

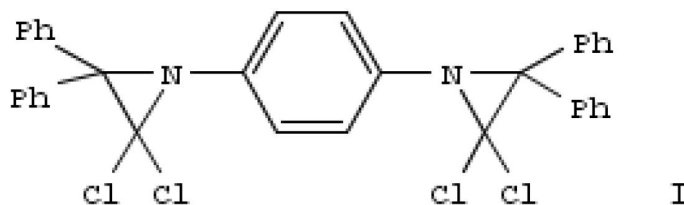
The adhesion of PVC [9002-86-2] coatings to polyester fabrics was improved by pretreating the fabric with a soln. of polyethylenimine (I) [9002-98-6] and, optionally, drying. Thus, a 200 g/cm² polyester fabric having 9 threads/cm for both warp and weft was treated with 1% (based on thread wt.) spin finish contg. 0.5% I. The pretreated fabric was coated with 100 g/m² mixt. of PVC paste 60, DOP 40, basic Pb carbonate 1.2, Ba Cd laurate 0.6, 70% soln. of OH-contg. polyester in 1:1 EtOAc-CH₂Cl₂ 6, and 50% TDI dimer [26747-90-0] in DOP 6.1 parts. After curing 1 min at 140° and 2 min at 190° , the coating had adhesion 142 N/5 cm, compared to 95 for untreated fabric.

66. Reactive additives for poly(ethylene terephthalate). I. Preparation of ketenimines

By Sikkema, D. J.; Molenaar, E.; Van Guldener, D. B.

From *Recueil des Travaux Chimiques des Pays-Bas* (1976), 95(6), 154-6. Language: English, Database: CAPLUS

Reaction of Ph₂CO with p-C₆H₄(NH₂)₂ in xylene at reflux in the presence of BF₃.Et₂O gave p-C₆H₄(N:CPh₂)₂ (82% yield), which was treated with CHCl₃-aq. NaOH soln. in the presence of PhCH₂N+Et₃Cl⁻, and the resultant aziridine I (62% yield) was dechlorinated with NaI or Zn to give p-C₆H₄(N:C:Ph₂)₂ in 82-93% yield. Z(N:C:Ph₂)₂ [Z = (CH₂)₆, bis(4-phenyl)methane] were also prepd. The bisketenimines are useful as chain coupling agents for processing poly(ethylene terephthalate) into high-performance tyre-yarns (no data).



67. Proton bonding to 1,8-bis(dialkylamino)naphthalenes

By De Groot, R. L.; Sikkema, D. J.

From *Recueil des Travaux Chimiques des Pays-Bas* (1976), 95(1), 10-14. Language: English, Database: CAPLUS
 Proton NMR spectra of 1,8-bis(dimethylamino)naphthalene (I and 1,8-bis(diethylamino)naphthalene solns. to which HOAc or F₃CCO₂H is added exhibit several characteristic features caused by proton exchange phenomena. In the presence of less than one mole of F₃CCO₂H per mole of diamine the protonated and unprotonated forms are observed separately. The proton in the protonated amine resonates at unusually low field (~20 ppm). When more F₃CO₂H is added another proton signal at low field, between 18 and 12 ppm downfield from TMS, is observed. The nature of the latter proton resonance is discussed. The proton in the amine is probably in a bridge between the two Ni atoms, since no magnetic inequivalence is observed for the four Me or Et groups attached to the N. In the presence of HOAc instead of F₃CCO₂H the proton in the amine molecule changes places with the other protons in the soln. From the temp. dependence of the spectra the activation energy for the exchange of D⁺ between amine mol. is estimated. The possible use of I as a probe for the investigation of H bonding phenomena in carboxylic acid solns. in nonaqueous solvents is indicated.